

Standard Operating Procedure**Title: Total Alkalinity - Titration Method****Purpose:**

To provide a standard operating procedure for calculating the total alkalinity of water samples following Standard Methods.

Scope:

This procedure applies to calculating the alkalinity of water samples only. The timing and frequency of this procedure will be determined by the study protocol.

Health/Safety Warning:

When working with potentially hazardous materials, follow EPA, OSHA, and other specific health and safety procedures. Be prepared in case of emergency (e.g., telephone numbers, first aid kit). Personnel should wear an apron or lab coat, gloves and safety glasses when handling hazardous chemicals. Consult MSDS before handling chemicals. Acids should be mixed under a fume hood (see SOP#GEN 102.0).

Equipment and Supplies:

Na₂CO₃ (CAS# 497-19-8; ex., Sigma #S-2127)

H₂SO₄ (CAS# 7664-93-9; ex., Sigma #S-1526)

Thermo Orion pH Meter Model 720A

Deionized (DI) Water

50 ml buret

Magnetic stir plate and stir bar

Sample Bottle

Reagents:

Standard Sodium Carbonate, 0.02 N: Dry a few grams of Na₂CO₃ at 140°C and cool in a desiccator. Boil deionized water to expel CO₂ and allow to cool. Add 1.06 g of the anhydrous Na₂CO₃ to a 1,000 ml volumetric flask and dilute to volume with the CO₂-free water. This solution must be used within a few hours of preparation.

Standard Sulfuric Acid Titrant, 0.02 N: Dilute 2.8 ml of H₂SO₄ to 1,000 ml with CO₂-free water in a volumetric flask. This solution is approximately 0.1 N. Dilute 200 ml of 0.1 N H₂SO₄ to 1,000 ml with CO₂-free water.

Standardize the Sulfuric Acid titrant by adding 10 ml of 0.02 N Standard Sodium Carbonate and 90 ml CO₂-free water into a 250 ml beaker. Titrate with sulfuric acid according to the pH endpoint procedure below and use the following equation to determine the normality of the sulfuric acid solution.

$$NV = N'V'$$

N is the normality of the standard
V is the volume of the standard (ml)
N' is the normality of the titrant
V' is the volume of the titrant (ml)

The standardized Sulfuric Acid titrant should be approximately 0.02N. If higher, this may be adjusted to 0.02N and confirmed as above. Store in a dated and labeled glass bottle; standardized Sulfuric Acid titrant can be used for 1 year.

No mention of reagent stability can be found. This infers indefinite stability. Preventing evaporation by storing in a capped container should keep solution stable indefinitely.

Standard Operating Procedure**Procedure:**

1. Using a clean sample bottle, collect sufficient sample from the water source to perform the required analyses; measure alkalinity within 24 hrs. Label each sample bottle using the date and related study identifier.
2. Before using the DI system, check the resistance on the digital readout of the system (located in electrical equipment room #111). Record resistance as required. Standard resistance for Type II DI water is >1. If there is an error message on the screen, contact the maintenance department. (See SOP #GEN 101.1).
3. Use a balance to weigh chemical for the reagent preparation. Accuracy of the balance should be checked daily before use with a calibration weight set (see SOP #GEN 103.0).
4. Prepare reagents as above (if needed).
5. Measure a 100 ml water sample into a 250 ml beaker. Set the sample on a magnetic stir plate with a stir bar.
6. Using a calibrated pH meter (see SOP #GEN 109.0), begin titration with the sulfuric acid solution to an end point of pH 4.5. When nearing the end point, slow down the titration rate and be sure that pH equilibrium is reached before adding more titrant.
7. Calculate the total alkalinity and record.

Calculations/Data Handling/Documentation:

Total Alkalinity (mg/L) = {(ml of titrant)(N)(50)(1,000)} / ml of sample

*If N = 0.02 and ml of sample = 100, then Total alkalinity (mg/L) = (ml of titrant)(10)

pH meter calibration and total alkalinity must be recorded in the appropriate log (ex., pH Meter Calibration log and Water Chemistry log, respectively).

References:

Boyd, Claude E. and Tucker, Craig S. 1992. Water Quality and Pond Soil Analyses for Aquaculture. Auburn University, Alabama.

APHA (American Public Health Association), American Water Works Association, and Water Pollution Control Federation. 2005. Standard Methods for the Examination of Water and Wastewater, 21st edition. Washington, D.C.

USDA/ARS- SNARC - SOP# GEN 102.0: Fume Hood.

USDA/ARS- SNARC - SOP# GEN 103.0: AT261 Balance.

USDA/ARS- SNARC - SOP# GEN 109.0: 720A pH Meter.


USDA/ARS - Stuttgart/Pine Bluff Location – Safety Health and Security Plan.

Standard Operating Procedure

Barker, K. 1998. At the Bench: a Laboratory Navigator. Cold Spring Harbor Laboratory Press. 460 pp.


Prepared by

10/15/2008
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10/15/2008
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